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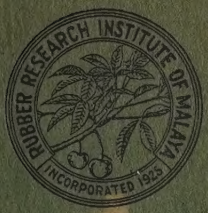

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THE PREPARATION OF SOFTENED RUBBER

As a result of work carried out at the laboratories of The Rubber Research Institute of Malaya three United Kingdom patents have been granted for inventions communicated by Jack Douglas Hastings and by Victor Harold Wentworth, both members of the staff of The Rubber Research Institute of Malaya.

The specifications of the three patents nos. 531,723; 535,837; 536,301 are as under:—

PATENT SPECIFICATION 531,723

Application Date: July 31, 1939. No. 22153/39.

Complete Specification Left: May 31, 1940

Complete Specification Accepted: January 9, 1941

PROVISIONAL SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3, (a communication from Jack Douglas Hastings, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of softened rubber with more especial reference to processes for the preparation of soft rubber by the incorporation of peptising agents into latex prior to coagulation so that the rubber is subsequently softened as a result of the effect of the heat applied during drying.

Peptising agents are well-known in the rubber industry for incorporating with rubber on the mill to facilitate processing and generally with a view to obtaining a softened product, and fall into two classes (a) Those which are toxic such as compounds of phenyl-hydrazine, and (b) Those which are non-toxic or substantially so such as thio-beta-naphthol or naphthyl-beta-mercaptan, and xylyl mercaptan. For estate use, owing to the health hazard, only the non-toxic peptising agents can be recommended.

In accordance with the present invention the incorporation of the peptising agent in the latex prior to coagulation is effected by first dispersing or dissolving the peptising agent in a liquid compatible with latex whereby the dispersion or solution may be thoroughly disseminated throughout the latex, then coagulating the latex and subsequently drying the coagulum to obtain a softened rubber.

The common organic volatile solvents for peptising agents, for example, benzene or acetone, have the advantage that they may easily be removed after coagulation but they are themselves coagulants of latex so that the addition of such solution is liable to cause local clotting preventing uniform distribution of the softening agent; moreover, the high rate of evaporation necessitates care being taken to avoid deposition of the peptising agent within the latex.

Satisfactorily uniform incorporation of the peptising agent thio-beta-naphthol or naphthyl-beta-mercaptan with latex has been obtained by emulsifying benzene solutions of the agent in water using potassium oleate as a stabiliser, the emulsion being worked up on a mill or homogeniser or in other convenient manner.

Various peptising agents are already marketed dissolved in inert hydrocarbon, and in the case of xylyl mercaptan so dissolved while the liquid peptiser is not miscible with water or latex it may be worked up into an aqueous emulsion with ease before being added to the latex and distributed therein.

In the practice of the invention according to one mode of carrying it into effect a softening emulsion is made by dissolving 10 gms. of thio-beta-naphthol in 100 ccs. of benzene, adding this to 900 ccs. of water containing 2 gms. of potassium oleate, and passing the mixture one or more times through a paste mill or homogeniser.

The emulsion while still fresh (as it has a tendency to cream when left) is then added to the latex immediately prior to coagulation with formic or acetic acid, the coagulum being drained and sheeted before being dried in a smoke house at a temperature of 120—140°F.

Addition of the emulsion to the latex does not affect coagulation in any way and no difficulty is experienced in sheeting the coagulum.

A suitable emulsion for mixing with latex may also be prepared without a paste mill or homogeniser by using an emulsifying agent such as that sold under the Registered Trade Mark Agral W.B.S. which is soluble in thio-beta-naphthol, and the cost of this emulsion can be still further reduced by dissolving the peptising agent and

emulsifying agent in kerosene instead of in a more volatile solvent such as benzene.

Progressive softening occurs as the amount of peptising agent added to the latex is increased, and higher drying temperatures, at least during the early stages, have been found to produce softer rubbers, an initial temperature of at least 140°F. during the early stages of drying and a lower temperature in the vicinity of 120°F. during the later stages having been found suitable.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

Dated this 31st day of July, 1939.

O'DONNELL, LIVSEY & CO.,

Chartered Patent Agents,
47, Victoria Street, Westminster,
London, S.W.1,

Agents for Applicants.

COMPLETE SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3, (a communication from Jack Douglas Hastings, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of softened rubber with more especial reference to improved processes for the preparation of soft rubber by the incorporation of peptising agents into latex prior to coagulation so that the rubber is subsequently softened as a result of the effect of the heat applied during drying.

Peptising agents are well known in the rubber industry for incorporating with rubber on the mill to facilitate processing and

generally with a view to obtaining a softened product, and fall into two classes (a) Those which are toxic such as compounds of phenyl-hydrazine, and (b) Those which are non-toxic or substantially so such as the mercaptonaphthalenes thio-beta-naphthol or naphthyl-beta-mercaptan, and xylyl mercaptan. For estate use, owing to the health hazard, only the non-toxic peptising agents can be recommended. Other substances such as mercapto-benzothiazole or nitroso compounds in aqueous solution or dispersion and referred to as peptisers or peptising agents have been proposed for addition to latex prior to coagulation with acid in the preparation of tacky or sticky rubber.

In accordance with the present invention the incorporation of mercapto-naphthalenes and xylyl mercaptans as peptising agent in the latex prior to coagulation is effected by first dispersing or dissolving the peptising agent in a liquid compatible with latex whereby the dispersion or solution may be thoroughly disseminated throughout the latex, then coagulating the latex and subsequently drying the coagulum to obtain a softened rubber.

The common organic volatile solvents for peptising agents, for example, benzene or acetone, have the advantage that they may easily be removed after coagulation but they are themselves coagulants of latex so that the addition of such a solution is liable to cause local clotting preventing uniform distribution of the softening agent; moreover, the high rate of evaporation necessitates care being taken to avoid deposition of the peptising agent within the latex.

Satisfactorily uniform incorporation of the peptising agent thio-beta-naphthol or naphthyl-beta-mercaptan with latex has been obtained by emulsifying benzene solutions of the agent in water using potassium oleate as a stabilizer, the emulsion being worked up on a mill or homogeniser or in other convenient manner.

Various peptising agents are already marketed dissolved in inert hydrocarbon, and in the case of xylyl mercaptan so dissolved while the liquid peptiser is not miscible with water or latex it may be worked up into an aqueous emulsion with ease before being added to the latex and distributed therein.

In the practice of the invention according to one mode of carrying it into effect a softening emulsion is made by dissolving 10 gms. of thio-beta-naphthol in 100 ccs. of benzene, adding this to 900 ccs. of water containing 2 gms. of potassium oleate, and passing the mixture one or more times through a paste mill or homogeniser.

The emulsion while still fresh (as it has a tendency to cream when left) is then added to the latex immediately prior to coagula-

tion with formic or acetic acid, the coagulum being drained and sheeted before being dried in a smoke house at a temperature of 120—140°F.

Addition of the emulsion to the latex does not affect coagulation in any way and no difficulty is experienced in sheeting the coagulum.

A suitable emulsion for mixing with latex may also be prepared without a paste mill or homogeniser by using an emulsifying agent which is soluble in the solution to be emulsified and forms an emulsion when treated with water containing a little alkali. Such an emulsifying agent is that sold under the Registered Trade Mark Agral W.B.S. which is soluble in thio-beta-naphthol, and the cost of this emulsion can be still further reduced by dissolving the peptising agent and emulsifying agent in kerosene instead of in a more volatile solvent such as benzene.

Progressive softening occurs as the amount of peptising agent added to the latex is increased, and higher drying temperatures, at least during the early stages, have been found to produce softer rubbers, an initial temperature of at least 140°F. during the early stages of drying and a lower temperature in the vicinity of 120°F. during the later stages having been found suitable.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the manufacture of softened rubber by the incorporation of mercaptonaphthalenes and xylyl mercaptans as peptising agents in latex prior to coagulation, consisting in dispersing or dissolving the peptising agent in a liquid compatible with latex, disseminating the dispersion or solution throughout the latex, then coagulating the latex and subsequently drying the coagulum to obtain a softened rubber.

2. A process for the manufacture of softened rubber according to Claim 1, wherein the peptising agent is dissolved in an inert hydrocarbon and the liquid peptiser is emulsified with an aqueous medium before being added to the latex and distributed therein.

3. A process for the manufacture of softened rubber according to either of the preceding Claims wherein the peptising agent is thio-beta-naphthol or naphthyl-beta-mercaptan dissolved in benzene and wherein the aqueous emulsion is worked up using a stabiliser such as potassium oleate.

4. A process according to any of the preceding Claims wherein the softening emulsion is made by dissolving thio-beta-naphthol in the proportion of 10 gms. to 100 ccs. of benzene, adding this to 900 ccs. of water containing 2 gms. of potassium oleate and passing the mixture one or more times through a paste mill or homogeniser.

5. A process according to any of the preceding Claims wherein the latex is coagulated with formic or acetic acid and the coagulum is drained and sheeted before being dried at 120°—140°F.

6. A process according to any of the preceding Claims wherein the initial drying of the coagulum is effected at a temperature of at least 140°F., the drying temperature being reduced in the later stages to about 120°F., and a softer rubber being thus obtained.

7. A process according to any of the preceding Claims wherein stabilisers or emulsifying agents are added to the peptiser and/or to the latex.

8. A process for the manufacture of softened rubber according to any of the preceding Claims substantially as described.

9. Softened rubber when prepared by the process according to any of the preceding Claims substantially as described.

Dated this 31st day of May, 1940.

O'DONNELL, LIVSEY & CO.,

Chartered Patent Agents,
47, Victoria Street, Westminster, S.W.1,

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PATENT SPECIFICATION 535,837

Application Date: December 1, 1939. No. 31245/39
Complete Specification Left: November 7, 1940
Complete Specification Accepted: April 23, 1941

PROVISIONAL SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3 (a communication from Victor Harold Wentworth, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of softened rubber with more especial reference to its preparation by the incorporation of peptising agents into latex prior to coagulation, whereby the rubber is subsequently softened as a result of the effect of heat applied during the drying process.

The use of peptising agents which are usually incorporated with the rubber on the mill in order to facilitate processing and also to produce a softened product is well known in the rubber industry. The chemical compositions of peptising agents fall into widely differing classes.

Coagulation of latex on the plantations is usually effected by the addition of an acid to the latex. The resultant coagulum is rolled thin while being sprayed with water and is then dried, usually at an elevated temperature. To ensure that a peptising agent shall remain in the rubber in order to exert its full effect during the drying process, it is preferable that it should be substantially insoluble in water whereby its removal from the coagulum during the rolling and washing operations is prevented.

In accordance with the present invention, peptising agents, of the class known as organic peroxides represented by the formula $R_1—CO—O—O—CO—R_2$, in which R_1 and R_2 may be aliphatic or aromatic radicals, are added to latex as emulsions in suitable

media. Coagulation of the latex, with simultaneous breakdown of the peptiser emulsion is brought about by known coagulants, such as formic acid or sodium silicofluoride. The coagulum, containing the peptiser, can subsequently be converted into sheet or crepe rubber in the usual manner, as required.

A peptising agent of the class described is benzoyl peroxide, which is usually marketed as a mixture having about a 30% water content. In the practice of the invention according to one mode of carrying it into effect, 230.4 grams of wet benzoyl peroxide (analysis 32.12% water) is mixed with 672 c.c. of benzene and 384 c.c. of a 2½% solution of potassium oleate. The mixture is vigorously agitated in a shaking machine and is then converted into an emulsion of the required concentration and stability by passing it through an homogeniser together with 6880 c.c. of water. This emulsion is added to latex having a dry rubber content of 15.29% in the proportion of 200 c.c. per gallon and is well mixed. Coagulation and simultaneous precipitation of the peptiser is produced by the addition of 8 fluid ounces of 1% (by volume) formic acid solution per gallon of latex.

The coagulum is passed through a sheeting machine and the resulting sheets are drained and dried. Sheets are also made from identical latex without the addition of the peptiser and are dried under the same conditions. The results of plasticity determinations made on both the treated and untreated sheets are shown in Table I.

Plasticity is expressed as the D_{30} value, a low value indicating a soft rubber. The D_{30} value is the thickness in mm. to which a spherical pellet weighing 0.4 gram is compressed in 30 minutes under a weight of 5 Kg. at a temperature of 100°C. in a parallel plate plastometer.

TABLE I

Conditions of drying	Rubber with 0.57% Benzoyl peroxide. D_{30} value	Rubber without peptiser. D_{30} value
In air at 140°F.	0.84	1.52
In a " tunnel " type Smoke-house	1.18	1.39

Progressive softening occurs as the amount of peptising agent added to the latex is increased and also with increased drying temperatures, as shown in Table II.

TABLE II

Benzoyl peroxide (on rubber) Per cent.	D_{30} values	
	Dried at 120° F. in air	Dried at 140° F. in air
0	1.52	1.54
0.17	1.45	1.31
0.34	1.11	0.89
0.67	0.63	0.43
1.00	0.41	0.21

It has been found that the softness of the rubber is markedly dependent on the conditions of drying in the initial stages. When drying normal sheet rubber, smoke-drying produces a softer sheet than air-drying but the reverse is the case when a peptiser such as benzoyl peroxide is present. This effect is shown by the D_{30} values in Table III. Identical sheet containing 0.6% benzoyl peroxide was dried in four days by combinations of air-drying and smoke-drying treatments. Irrespective of temperature, a harder rubber resulted from smoke-drying on the first day.

TABLE III

Conditions of drying	Number of days, in order from top to bottom							
Hot air @ 120°F.	1		1					
Hot air @ 140°F.					1		1	
"Tunnel" Smoke-house, hot end, approx. 145°F. ...		1				1		
"Tunnel" Smoke-house, cool end, approx. 125°F. ...				1				1
Hot air @ 120°F.		3		3				
Hot air @ 140°F.						3		3
"Tunnel" Smoke-house, hot end, approx. 145°F. ...	3				3			
"Tunnel" Smoke-house, cool end, approx. 125°F. ...			3				3	
D_{30} value	0.57	1.12	0.64	1.19	0.71	1.09	0.59	1.26

If it is desired to prepare smoked sheet and also secure the utmost softening from a given amount of peptiser, it is preferable to submit the sheets to an initial drying period in hot air, free from smoke, followed by the required amount of smoking.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

Dated the 1st day of December, 1939.

O'DONNELL, LIVSEY & CO.,

Chartered Patent Agents,
47, Victoria Street, Westminster,
London, S.W.1,

Agents for Applicants.

COMPLETE SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3 (a communication from Victor Harold Wentworth, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of softened rubber by the incorporation of peptising agents into latex prior to coagulation, whereby the rubber is subsequently softened as a result of the effect of heat applied during the drying process.

The use of peptising agents which are usually incorporated with the rubber on the mill in order to facilitate processing and also to produce a softened product is well known in the rubber industry. The chemical compositions of peptising agents fall into widely differing classes.

Coagulation of latex on the plantations is usually effected by the addition of an acid to the latex. The resultant coagulum is rolled thin while being sprayed with water and is then dried, usually at an elevated temperature. To ensure that a peptising agent shall remain in the rubber in order to exert its full effect during the drying process, it is preferable that it should be substantially insoluble in water whereby its removal from the coagulum during the rolling and washing operations is prevented.

In accordance with the present invention, peptising agents, of the class known as organic peroxides represented by the formula $R_1—CO—O—O—CO—R_2$, in which R_1 and R_2 may be aliphatic or aromatic radicals, are added to latex as emulsions in suitable media. Coagulation of the latex, with simultaneous breakdown of the peptiser emulsion is brought about by known coagulants, such as formic acid or sodium silicofluoride. The coagulum, containing the peptiser, can subsequently be converted into sheet or crepe rubber in the usual manner, as required.

A peptising agent of the class described is benzoyl peroxide ($C_{14}H_{10}O_4$ or $C_6H_5—CO—O—O—CO—C_6H_5$) which is usually marketed as a mixture having about a 30% water content and appropriate proportions are from 0.5 to 1.0% by weight of the rubber. In the practice of the invention according to one mode of carrying it into effect, 230.4 grams of wet benzoyl peroxide (analysis 32.12% water) is mixed with 672 c.c. of benzene and 384 c.c. of a 2½% solution of potassium oleate. The mixture is vigorously agitated in a shaking machine and is then converted into an emulsion of the required concentration and stability by passing it through an homogeniser together with 6880 c.c. of water. This emulsion is added to latex having a dry rubber content of 15.29% in the proportion of 200 c.c. per gallon and is well mixed. Coagulation and simultaneous precipitation of the peptiser is produced by the addition of 8 fluid ounces of 1% (by volume) formic acid solution per gallon of latex.

The coagulum is passed through a sheeting machine and the resulting sheets are drained and dried at about 120°—140°F. Sheets are also made from identical latex without the addition of the peptiser and are dried under the same conditions. The results of plasticity determinations made on both the treated and untreated sheets are shown in Table I.

Plasticity is expressed as the D_{30} value, a low value indicating a soft rubber. The D_{30} value is the thickness in mm. to which a spherical pellet weighing 0.4 gram is compressed in 30 minutes under a weight of 5 Kg. at a temperature of 100°C. in a parallel plate plastometer.

TABLE I

Conditions of drying	Rubber with 0.57% Benzoyl peroxide. D_{30} value	Rubber without peptiser. D_{30} value
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In a " tunnel " type Smoke-house	1.18	1.39

Progressive softening occurs as the amount of peptising agent added to the latex is increased and also with increased drying temperatures, as shown in Table II.

TABLE II

Benzoyl peroxide (on rubber) Per cent.	D_{30} values	
	Dried at 120° F. in air	Dried at 140° F. in air
0	1.52	1.54
0.17	1.45	1.31
0.34	1.11	0.89
0.67	0.63	0.43
1.00	0.41	0.21

It has been found that the softness of the rubber is markedly dependent on the conditions of drying in the initial stages. When drying normal sheet rubber, smoke-drying produces a softer sheet than air-drying but the reverse is the case when a peptiser such as benzoyl peroxide is present. This effect is shown by the D_{30} values in Table III. Identical sheet containing 0.6% benzoyl peroxide was dried in four days by combinations of air-drying and smoke-drying treatments. Irrespective of temperature, a harder rubber resulted from smoke-drying on the first day.

If it is desired to prepare smoked sheet and also secure the utmost softening from a given amount of peptiser, it is preferable to submit the sheets to an initial drying period in hot air at 120°F., free from smoke, followed by the required amount of smoking.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

TABLE III

Conditions of drying	Number of days, in order from top to bottom							
Hot air @ 120°F.	1		1					
Hot air @ 140°F.					1		1	
"Tunnel" Smoke-house, hot end, approx. 145°F. ...		1				1		
"Tunnel" Smoke-house, cool end, approx. 125°F. ...				1				1
Hot air @ 120°F.		3		3				
Hot air @ 140°F.						3		3
"Tunnel" Smoke-house, hot end, approx. 145°F. ...	3				3			
"Tunnel" Smoke-house, cool end, approx. 125°F. ...			3				3	
D_{30} value	0.57	1.12	0.64	1.19	0.71	1.09	0.59	1.26

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the preparation of softened rubber consisting in adding to latex emulsions of organic peroxides of the formula $R_1—CO—O—O—CO—R_2$ in which R_1 and R_2 may be aliphatic or aromatic radicals, coagulating the latex and simultaneously breaking down the peptiser or softening emulsion so that the peroxide is deposited in the coagulum.

2. A process according to claim 1 wherein benzoyl peroxide is employed and the coagulation and simultaneous breakdown of the softening emulsion is effected by the addition of formic acid or sodium silicofluoride.

3. A process according to either of the preceding claims wherein the coagulum is sheeted or creped and then dried at about 120°—140°F.

4. A process according to any of the preceding claims wherein the proportion of peptiser is from 0.5 to 1% by weight of the rubber.

5. A process according to any of the preceding claims wherein benzoyl peroxide of about 30% water content and in the proportion of 230 grams is mixed with 672 c.c. of benzene and 384 c.c. of a 2½% solution of potassium oleate, the mixture being vigorously agitated and emulsified with 6880 c.c. of water before being added to the latex.

6. A process according to claim 5 wherein for latex having a dry rubber content of 15% the proportion of the emulsion is 200 c.c. per gallon.

7. A process according to any of the preceding claims wherein the coagulum is initially dried in air at 120°F. followed by the required amount of smoking if smoked sheet is to be prepared, thus to obtain a softer rubber.

8. A process for the preparation of softened rubber such as described.

9. Softened rubber when prepared by the process according to any of the preceding claims such as described.

Dated this 7th day of November, 1940.

O'DONNELL, LIVSEY & CO.,

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47, Victoria Street, Westminster,
London, S.W.1,

Agents for Applicants.

PATENT SPECIFICATION 536,301

Application Date: December 1, 1939. No. 31244/39
Complete Specification Left: November 7, 1940
Complete Specification Accepted: May 9, 1941

PROVISIONAL SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3 (a communication from Victor Harold Wentworth, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of softened rubber with more especial reference to its preparation by the incorporation of peptising agents into latex prior to coagulation whereby the rubber is subsequently softened as a result of the effect of heat applied during the drying process.

The use of peptising agents which are usually incorporated with the rubber on the mill in order to facilitate processing and also to produce a softened product is well known in the rubber industry. The chemical compositions of peptising agents fall into different classes with the result that, in addition to their effect in softening the raw rubber, some have an influence on the subsequent vulcanisation process, whilst others are inert.

The coagulation of latex on the plantations is usually effected by the addition of an acid to the latex. The resultant coagulum is rolled thin while spraying with water and then dried usually at an elevated temperature. To ensure that a peptising agent shall remain in the rubber in order to exert its full effect during the drying process, it is preferable that it should be substantially insoluble in water whereby its removal from the coagulum during the rolling and washing operations is prevented.

In accordance with the present invention peptising agents which are insoluble in water but possess an acidic grouping capable of reacting with an alkali to form a soluble salt, are added to latex as a solution in an alkaline medium. Coagulation of the latex is produced by the addition of an acid and simultaneously the peptising agent is precipitated in intimate contact with the coagulated rubber. The coagulum can subsequently be converted into sheet or crepe rubber in the usual manner, as required.

Several chemical groupings which possess acidic hydrogen or confer solubility in alkaline solutions, are known to those versed in the art and include carboxy, phenolic and mercapto groups.

An acidic peptising agent is conveniently prepared in a form suitable for its addition to latex by dissolving it either in strong caustic soda or potash solution and then suitably adjusting its concentration before adding to the latex. The requisite amount of peptiser solution is stirred thoroughly into the latex and coagulation is produced by the addition of acid, using a quantity sufficient to ensure the simultaneous precipitation of the peptiser. Mercaptobenzthiazole is a suitable acidic peptising agent.

In the practice of the invention, according to one mode of carrying it into effect, 140 grams of mercaptobenzthiazole are dissolved in 312 c.c. of 2.89 N. caustic soda solution and diluted to 7000 c.c. with water. This solution is added to latex having a dry rubber content of 15.63%, in the proportion of 170 c.c. per gallon and is well mixed. Coagulation and simultaneous precipitation of the peptising agent is produced by the addition of 1000 c.c. of 0.25% (by volume) formic acid solution to each gallon of latex. It has been found that, in the presence of the peptiser, smoother coagulation is achieved with formic acid of 0.25% strength rather than with 1% strength. Normal field latex diluted to a D.R.C. of 15% is coagulated with approximately 200 c.c. of 1% formic acid solution.

The coagulum is passed through a sheeting machine and the resulting sheets are drained and dried. Sheets are also made from the same latex without the addition of the peptiser and are dried under identical conditions. The results of plasticity determinations on both the treated and untreated samples are shown in Table I.

Plasticity is expressed as the D_{30} value, a low value indicating a soft rubber. The D_{30} value is the thickness in mm. to which a spherical pellet weighing 0.4 gram is compressed in 30 minutes under a weight of 5 Kg. at a temperature of 100°C. in a parallel plate (Williams) plastometer.

TABLE I

Conditions of drying	Rubber with 0.48% Mercaptobenzthiazole. D_{30} value	Rubber without peptiser. D_{30} value
In air at 140°F.	0.63	1.52
In a "tunnel" type smoke-house	0.80	1.33

Progressive softening occurs as the amount of peptising agent added to the latex is increased and also with increased drying temperatures, as shown in Table II.

TABLE II

Mercaptobenzthiazole (on rubber) per cent.	D_{30} values	
	Dried at 120° F. air	Dried at 140° F. air
0	1.52	1.54
.18	1.42	1.23
.35	1.07	0.96
.70	0.64	0.50
1.05	0.54	0.26

In another experiment the following results were obtained.

TABLE III

Mercaptobenzthiazole (on rubber) per cent.	D_{30} values at four drying temperatures			
	120° F.	140° F.	160° F.	180° F.
0	1.65	1.63	1.52	1.60
0.62	0.58	0.43	0.42	0.31

It has been found that the softness of the rubber is markedly dependent on the temperature of drying in the initial stages. The later stages of drying may be at a reduced temperature without appreciable reduction of softness.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

Dated this 1st day of December, 1939.

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COMPLETE SPECIFICATION

Improvements in and relating to the Preparation of Softened Rubber

We, The British Rubber Producers' Research Association, a Body Corporate duly organised under the Laws of Great Britain, of 19, Fenchurch Street, London, E.C.3 (a communication from Victor Harold Wentworth, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Federated Malay States), do hereby declare the nature of this invention, and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of softened rubber with more especial reference to its preparation by the incorporation of peptising agents into latex prior to coagulation whereby the rubber is subsequently softened as a result of the effect of heat applied during the drying process.

The use of peptising agents which are usually incorporated with the rubber on the mill in order to facilitate processing and also to produce a softened product is well known in the rubber industry. The chemical compositions of peptising agents fall into different classes with the result that, in addition to their effect in softening the raw rubber, some have an influence on the subsequent vulcanisation process, whilst others are inert.

The coagulation of latex on the plantations is usually effected by the addition of an acid to the latex. The resultant coagulum is rolled thin while spraying with water and then dried usually at an elevated temperature. To ensure that a peptising agent shall remain in the rubber in order to exert its full effect during the drying process, it is preferable that it should be substantially insoluble in water whereby its removal from the coagulum during the rolling and washing operations is prevented.

In accordance with the present invention peptising agents which are insoluble in water but possess an acidic grouping capable of reacting with an alkali to form a soluble salt, are added to latex as a solution in an alkaline medium. Coagulation of the latex is produced by the addition of an acid and simultaneously the peptising agent is precipitated in intimate contact with the coagulated rubber. The coagulum can subsequently be converted into sheet or crepe rubber in the usual manner, as required.

Several chemical groupings which possess acidic hydrogen or confer solubility in alkaline solutions, are known to those versed in the art and include carboxy, phenolic and mercapto groups.

An acidic peptising agent is conveniently prepared in a form suitable for its addition to latex by dissolving it either in strong caustic soda or potash solution and then suitably adjusting its concentration before adding to the latex. The requisite amount of peptiser solution corresponding to about 0.5 to 1% by weight of the rubber is stirred thoroughly into the latex and coagulation is produced by the addition of acid, using a quantity sufficient to ensure the simultaneous precipitation of the peptiser. Mercaptobenzthiazole is a suitable acidic peptising agent.

In the practice of the invention, according to one mode of carrying it into effect, 140 grams of mercaptobenzthiazole are dissolved in 312 c.c. of 2.89 N. caustic soda solution and diluted to 7000 c.c. with water. This solution is added to latex having a dry rubber content of 15.63%, in the proportion of 170 c.c. per gallon and is well mixed. Coagulation and simultaneous precipitation of the peptising agent is produced by the addition of 1000 c.c. of 0.25% (by volume) formic acid solution to each gallon of latex. It has been found that, in the presence of the peptiser, smoother coagulation is achieved with formic acid of 0.25% strength rather than with 1% strength. Normal field latex diluted to a D.R.C. of 15% is coagulated with approximately 200 c.c. of 1% formic acid solution.

The coagulum is passed through a sheeting machine and the resulting sheets are drained and dried. Sheets are also made from the same latex without the addition of the peptiser and are dried under identical conditions. The results of plasticity determinations on both the treated and untreated samples are shown in Table I.

Plasticity is expressed as the D_{30} value, a low value indicating a soft rubber. The D_{30} value is the thickness in mm. to which a spherical pellet weighing 0.4 gram is compressed in 30 minutes under a weight of 5 Kg. at a temperature of 100°C. in a parallel plate (Williams) plastometer.

TABLE I

Conditions of drying	Rubber with 0.48% Mercaptobenzthiazole. D_{30} value	Rubber without peptiser. D_{30} value
In air at 140°F.	0.63	1.52
In a "tunnel" type smoke-house	0.80	1.33

Progressive softening occurs as the amount of peptising agent added to the latex is increased and also with increased drying temperatures, as shown in Table II.

TABLE II

Mercaptobenzthiazole (on rubber) per cent.	D_{30} values	
	Dried at 120° F. air	Dried at 140° F. air
0	1.52	1.54
.18	1.42	1.23
.35	1.07	0.96
.70	0.64	0.50
1.05	0.54	0.26

In another experiment the following results were obtained.

TABLE III

Mercaptobenzthiazole (on rubber) per cent.	D_{30} values at four drying temperatures			
	120° F.	140° F.	160° F.	180° F.
0	1.65	1.63	1.52	1.60
0.62	0.58	0.43	0.42	0.31

It has been found that the softness of the rubber is markedly dependent on the temperature of drying in the initial stages the

range 140°—180°F. being most suitable. The later stages of drying may be at a reduced temperature, say 110°—140°F., without appreciable reduction of softness.

Any appropriate stabilisers or emulsifying agents may be added to the peptiser or to the latex prior to addition, and the latex may be natural, diluted, preserved or concentrated, as will be understood.

By the present invention an improved, convenient and economical process for the preparation of soft rubber is obtained utilising existing estate equipment and eliminating the necessity for the installation of large and expensive plant.

We are aware that in the preparation of sticky compositions which may be subsequently vulcanised, it has been proposed to add to concentrated latex small proportions of mercaptothiazol peptising agents dissolved in alkaline medium, from the latex so treated to obtain deposits by dipping, spreading, spraying or electrophoresis, to dry the latex so deposited, and then to heat the deposits at about 100°C., and no claim is made herein to any such process.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the preparation of softened rubber consisting in adding as a solution in alkaline medium to latex a peptising agent insoluble in water but possessing an acidic grouping capable of reacting with the alkali to form a soluble salt, coagulating the latex with acid and thereby simultaneously precipitating the peptising agent in intimate contact with the coagulated rubber.

2. A process according to claim 1 wherein the peptising agent includes carboxy, phenolic or mercapto groups, e.g., mercaptobenzthiazole in the proportion of 0.5 to 1% by weight of the rubber, dissolved in caustic soda or potash.

3. A process according to either of the preceding claims wherein the coagulum is sheeted or creped and then dried initially at 120°—180°F.

4. A process according to any of the preceding claims wherein the initial drying is effected in air at 140°—180°F. followed by drying at a lower temperature of 110°—140°F. in smoke if smoked sheet is required.

5. A process according to any of the preceding claims wherein mercaptobenzthiazole in the proportion of 140 grams is dissolved in 312 c.c. of 2.89 N. caustic soda and diluted to 7000 c.c. with

water when the solution is added to latex in the proportion of 170 c.c. per gallon and will be mixed therein.

6. A process according to the preceding claim 5 wherein coagulation of the rubber and simultaneous precipitation of the peptising agent is effected by adding 1000 c.c. of 0.25% (by volume) formic acid solution to each gallon of latex.

7. A process for the preparation of softened rubber substantially as described.

8. Softened rubber when prepared by the process according to any of the preceding claims, substantially as described.

Dated this 7th day of November, 1940.

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